

**IN THE U.S. PATENT AND TRADEMARK OFFICE**

In re Application of: MICHALUK *et al.* )

APPLICATION NO.: 09/922,815 )

GROUP ART UNIT: 1742

Filed: August 6, 2001 )

Examiner: A. Oltmans

For: HIGH PURITY TANTALUM PRODUCTS CONTAINING THE SAME AND METHODS OF MAKING  
THE SAME

**DECLARATION UNDER 37 C.F.R. § 1.132**

Assistant Commissioner for Patents  
Washington, D.C. 20231

September 18, 2002

Sir:

1.) I, Christopher Michaluk, do declare and state as follows: I graduated from Drexel University in 1986 with a B.S. in Materials Engineering and received a master's degree in 1994 in Materials Engineering from Drexel University.

2.) Since December 1990, I have been employed by Cabot Corporation at its production, research, and development facility in Boyertown, Pennsylvania (Cabot Performance Materials), and during that time I have been engaged in research and development concerning metallurgy, including tantalum and products containing the same.

3.) I am one of the co-inventors of the above-identified patent application. I am familiar with the Office Action dated June 3, 2002, received in the above-identified application, as well as the references cited therein regarded by the Examiner as rendering the present invention obvious.

4.) This Declaration is submitted so that the Examiner can appreciate that the differences of the claimed invention over the technology of the cited references.

### **The Purity Levels of Douglass et al.**

5.) The Examiner relies upon Douglass et al. in the rejection and it is my understanding that the Examiner is taking the position that Douglass et al. shows any purity of tantalum which is mixed with yttrium to form a tantalum-yttrium alloy. As I explained in the interview with the Examiners, Douglass et al. does not mention at all the purity level of the tantalum used to make the alloy. It is my understanding that the Examiner believes that "pure tantalum" refers to a tantalum metal having no impurities whatsoever, in other words, 100% pure tantalum. However, I am unaware of any tantalum product either experimental grade or commercial grade that is 100% pure tantalum. To the best of my knowledge, no such purity level exists today or back in the 1960s during the time frame of Douglass et al.

6.) Since Douglass et al. does not explicitly mention the purity levels of the tantalum used and since the general understanding in metallurgy is that tantalum contains a certain amount of impurities, the impurity levels of the tantalum used by Douglass et al. must be the impurity levels that were generally present in tantalum during the 1960s. As an example, the article entitled, "The Effect of Yttrium on the Recrystallization and Grain Growth of Tantalum" by L. D. Kirkbride et al. (1965) shows a tantalum-yttrium alloy containing 14 ppm to 72 ppm yttrium. This material, as indicated in the article, was procured from National Research Corporation, the same Assignee of Douglass et al. This material falls squarely within Douglass et al. and thus is quite representative of the type of tantalum material that was present in the 1960s and was probably the type of tantalum used by Douglass et al. due to the fact that the material is from National Research Corporation and because of the time period. As can be seen at Table I, page 394, of this document, various tantalum-yttrium alloys are described but also provided in the same table are impurity levels. The Examiner will note that various impurities are provided for Nb (which is niobium), zirconium, molybdenum,

and other metals. The reference to the  $<$ , as discussed in the interview, simply means that this is the minimum detection limit "MDL" and one skilled in the art and the industry in general takes this number as the impurity present. In other words, the notation  $<100$  ppm has the impurity level of 100 ppm. To further assist the Examiner in this understanding, attached is an ASTM designation F2113-01, which indicates, for instance at section 4.2, that reported minimum detection limits are understood to have that element present at that minimum detection limit. From personal experience, this is the standard Cabot Corporation takes with all of its tantalum products as well as the customers that receive products from Cabot Corporation. As Mr. Gray, during the interview informed the Examiners, typically these amounts that are reported as "less than" generally means that a detection has occurred but that the detection device is unable to provide an exact ppm level other than the minimum detection level. Thus, looking at Table I of the Kirkbride article, it is clear that the tantalum alloy used in 1965 from National Research Corporation has metallic impurity levels exceeding 180 ppm which would make the product 99.982% pure which is clearly outside many claims of the claims of the present application reciting purity levels.

7.) In addition, attached is a press release dated July 2, 1964 from National Research Corporation, the same Assignee of the Douglass et al. patent relied upon by the Examiner. This paper discusses a tantalum product designated as SGS, for stable grain size. The data sheet for SGS tantalum that is referenced in the press release describes the chemical purity of the product. As can be seen, the typical ppm impurities exceeded 115 ppm with respect to metallic impurities and a more fair assessment shows other impurities approaching 100 ppm on a maximum level. At a minimum, the purity of the tantalum from this listing would 99.988% pure tantalum if one ignores "all others" impurities. Even taking this best case scenario, which I believe it not entirely reasonable, the purity level is still lower than the purity levels recited in claims that recite high

purity levels. These papers clearly show the type of purity levels that were present especially from National Research Corporation and provide, in my opinion, a fair assessment of the type of purity levels present in the tantalum used by Douglass et al. I am unaware of any purity levels existing on the order of 99.995% tantalum back in the 1960s from my understanding of the published papers and the stated technology in the 1960s.

8.) Accordingly, based on the attached documents, in my opinion, one skilled in the art, including myself, would consider the tantalum used in Douglass et al. to be a purity significantly below 99.995% tantalum.

**The Texture of Douglass et al.**

9.) In Douglass et al., a tantalum ingot is melted in the presence of yttrium to form a tantalum-yttrium alloy containing 10 to 1000 ppm yttrium. The ingot is reduced to plate stock of about ¼ inch thick and then subjected to an anneal of at least 3000° F and preferably 3400° to 3600° F to achieve substantially complete recrystallization of the tantalum alloy. Then, the tantalum alloy is rolled to a sheet with an intermediate anneal to achieve a thickness of .050 inch. The sheet is then rolled to .020 thickness and given a final anneal of 2200° F. The annealing, as stated in Douglass et al., is on the order of one hour. The examples of Douglass et al. are essentially consistent with this working of the metal at these temperatures. As stated in the interview with the Examiners, annealing first at temperatures above 3000° F, and for the annealing time used in Douglass et al. would produce a texture of a primary (100) texture. Unlike Douglass et al., in the preferred method of making the texture set forth in many of the claims of the present application, which is preferably a primary (111) uniform texture, the tantalum metal is flat forged into a rolling slab which is then annealed at a sufficient temperature and for a sufficient time to achieve at least partial recrystallization of the rolling slab. As shown in the examples set forth in the present application,

this temperature preferably does not exceed 1150° C which is quite different from the 3000° F annealing occurring in Douglass et al. Furthermore, in the preferred method of making the primary (111) uniform texture in the present application, the slab is then cold or warm rolled in both the perpendicular and parallel directions to the axis of the starting tantalum metal to form at least one plate wherein the plate is then flattened and subsequently annealed to achieve a certain grain size and texture. Looking at Douglass et al., there is one basic rolling of the tantalum alloy to form a sheet and there is absolutely no mentioning of cold or warm rolling in the perpendicular and parallel directions. Furthermore, after this rolling, the sheet is then given a final anneal at 2200° F in Douglass et al., and in the final anneal for the preferred method of the present application, the temperature is preferably kept at a temperature of 950° to about 1000° C which is lower than the final anneal temperatures referenced in Douglass et al. Thus, based on the manner in which the material is annealed as well as cold or warm rolled, the present invention achieves a preferred (111) texture while Douglass et al. would achieve essentially a primary (100) texture based on my understanding and experience with respect to the formation of textures.

#### **The Texture of Shah et al.**

10.) With respect to Shah et al., this patent specifically mentions that the texture achieved for the tantalum product has a uniform (100) texture which is the completely different and distinguishable from a primary (111) texture. Furthermore, many production details are not mentioned in Shah et al., for instance, annealing temperatures for the billet are not mentioned nor the annealing times. Thus, one has no choice but to rely on the (100) texture stated in Shah et al. which is clearly different from the primary (111) texture achieved in the present invention and in fact would even be different from a mixed (111/100) texture. Thus, one will note that unlike the preferred process of the claimed invention, Shah et al. does a conventional upset forging and rolling whereas in the

preferred process of the present invention, the present invention provides preferred annealing temperatures and times such as 950°-1100° F for final anneal temperatures involving a round processing. There are no final temperatures or times set forth in Shah et al. and one must assume that in view of the texture achieved, which was (100) across the face and through the thickness of the tantalum metal in Shah et al., that clearly different annealing temperatures were used as well as the time of anneal.

#### **The Purity of Shah et al.**

11.) As discussed during the interview, Shah et al. only provides the purity of one tantalum metal which has a purity of 99.95% which is explicitly set forth in Table 2 of Shah et al. No other purities are provided in Shah et al. As discussed during the interview, there are competing interests when high purity tantalum is used. In other words, at least three (3) important factors compete with each other, namely the purity of the tantalum, the grain size of the tantalum, and the texture of the tantalum. Just because a grain size is achieved for one purity level of tantalum does not mean this grain size will automatically be a given for a higher purer tantalum product and this also true for texture. Shah et al. strictly limited his study to 99.95% tantalum and thus, Shah et al. only provides this limited guidance to a person skilled in the art. Shah et al. essentially provides no information on what will happen and what needs to be done to achieve low grain sizes and desirable textures for higher purity products. In my opinion, Shah et al. only relates to a purity level of 99.95% and not to higher purity levels.

#### **The Activities of Cabot Corporation**

12.) Attached to the Information Disclosure Statement, are several sales by Cabot Corporation as well as a quote that was provided for a 99.999% pure tantalum product. I was the person involved in many of the sales that are set forth in the attached Supplemental Information Disclosure

Statement as well as the quote provided for 99.999% tantalum. To the best of my knowledge and recollection, the sales did not involve low grain sizes in combination with a high purity tantalum metal or in combination with a texture as described in the claims of the present application. The texture that would have been present, in my opinion, would have been a non-uniform mixed (100):(111) texture containing bands of primary (100) texture, due to the general information provided in the attached paperwork and my recollection of the processing used. The preferred process of the claimed invention is different from the process used to make the tantalum material set forth in Documents A-C. The quote in Document D that is set forth as part of the Information Disclosure Statement relates to a quote for a product that was not made at the time and did not involve any grain sizes or textures. It was simply for a high purity tantalum product without concern or consideration for grain size or texture. It was only after this time period and through research that a high purity tantalum product having desirable grain size and/or texture was achieved. Accordingly, in my opinion, none of the information provided in the Supplemental Information Disclosure Statement teaches or suggests the claimed invention as set forth in the present application.

13.) I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and any such willful false statement may jeopardize the validity of the application or any issuing thereon

Date:

9/18/02

  
Christopher Michalak